High-throughput additive manufacturing and characterization of HEAs for fusion applications

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Collaborators and Acknowledgements

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- NNSA ETI: DE-NA0003921 (NNSA Consortia)
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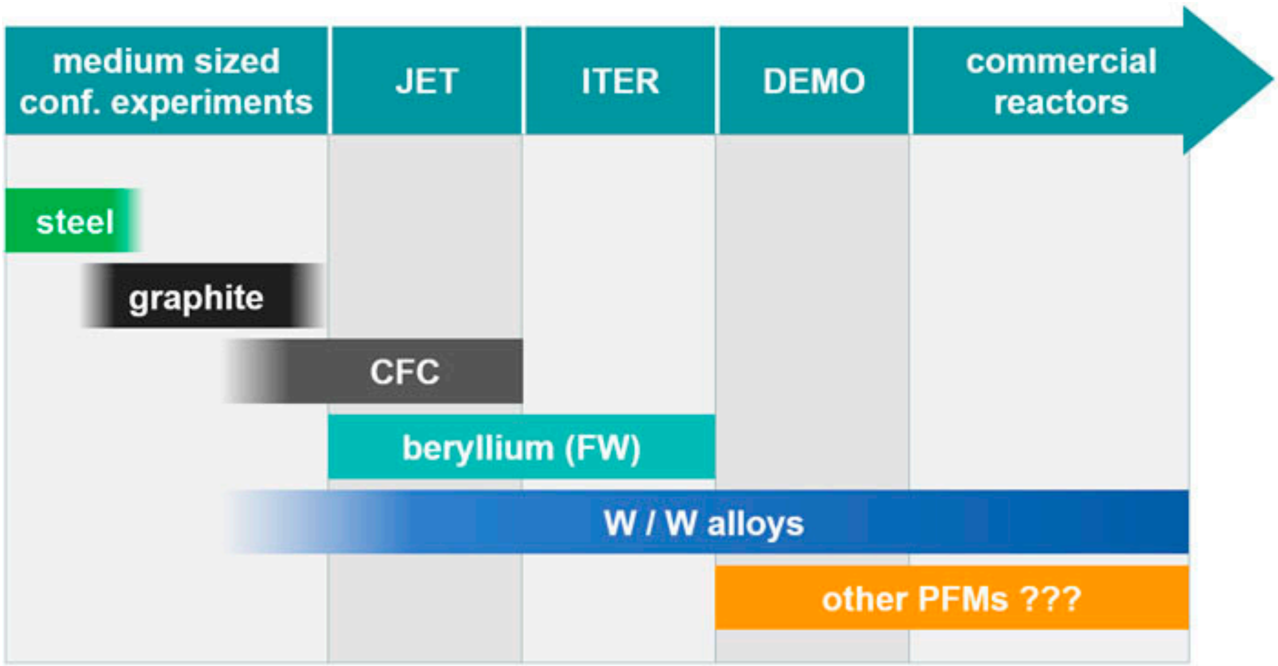
- ARPA-E: DE-AROOO1431 (ULTIMATE)

Objectives/Outline

- Materials to consider
- Advanced fabrication techniques for novel alloys
- Scale-up potential
- Challenges/opportunities at production level for rapid design

Materials

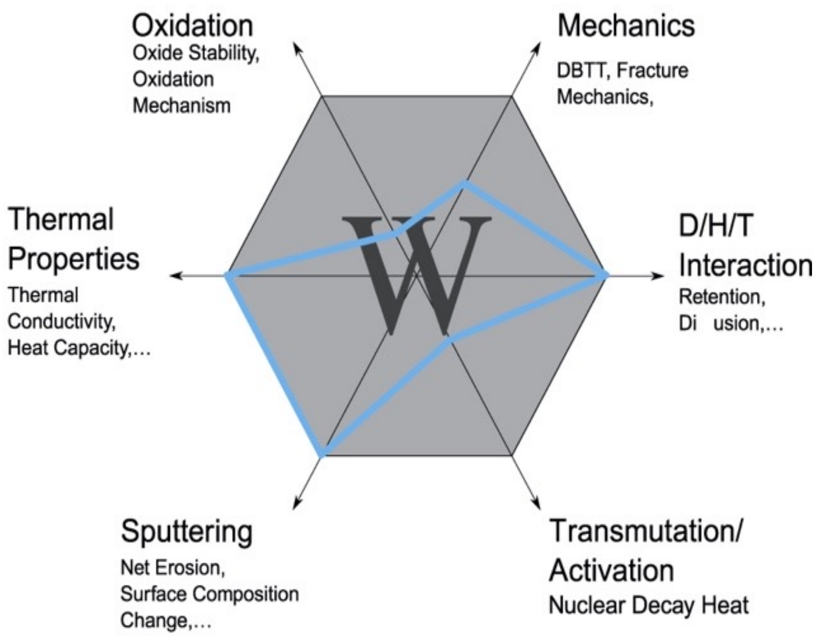
What are viable fusion materials?



Jochen Linke, et al. *Matter Radiat. Extremes* **4**, (2019) 056201

- **General:** oxide dispersion strengthened metals, refractories, ceramics, composites
- **Specific:** SiC, boron carbide, carbon fiber composites, tungsten, molybdenum
- A variety of multi-layer tiles that are combinations of above

Best possible plasma facing component (for debate)



Property	At Room Temperature (RT)	At 1300C[a]	At 20dpa (14MeV neutron equivalent) and RT	At 20dpa (14MeV neutron equivalent) and 1300C[a]
Yield Strength (MPa)	>250 MPa	>100 MPa	>250 MPa	>150 MPa
Ultimate Tensile Strength (MPa)	>350 MPa	>200 MPa	>350 MPa	>250 MPa
Failure Elongation (%)	>20%	>20%	>5%	>5%
Fracture Toughness (MPa $m^{1/2}$)	>50 MPa√m	>50 MPa√m	>10 MPa√m	>10 MPa√m
Creep Rupture Stress (MPa) @ 1000hr	NA	>80MPa	NA	>80MPa
Thermal Conductivity (W/mK)	>20 W/mK	>20 W/mK	>20 W/mK	>20 W/mK
Volumetric Swelling (%)	NA	NA	<2%	<2%
Neutron Sputtering Rate (µm/yr)	NA	NA	< 100 µm/yr	< 100 µm/yr
Fatigue Failure Cycles (N)	>50,000	>50,000	>10,000	>10,000
Total Activation Dose (on contact after 24hrs) - Rem	NA	NA	<5 Rem	<5 Rem

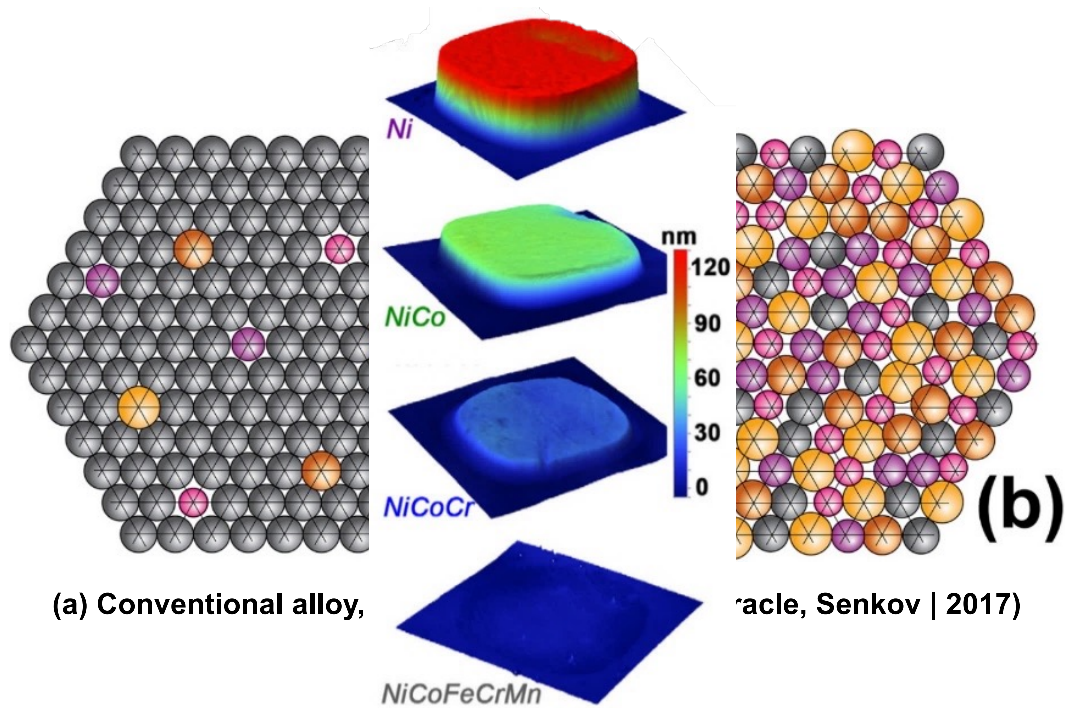
Strengths and weakness for W as PFC material

JW Coenen, et al., *Physical Scripta* (2016) 014002.

High-Entropy Alloy (HEA) overview

- High-entropy alloys (HEAs) are alloys with **multiple principal elements**
- Typically, no element >35 at%
- Usually defined as being primarily a solid-solution matrix

- Promising properties have been observed:
- High-temperature strength
- High specific strength
- Enhanced radiation tolerance

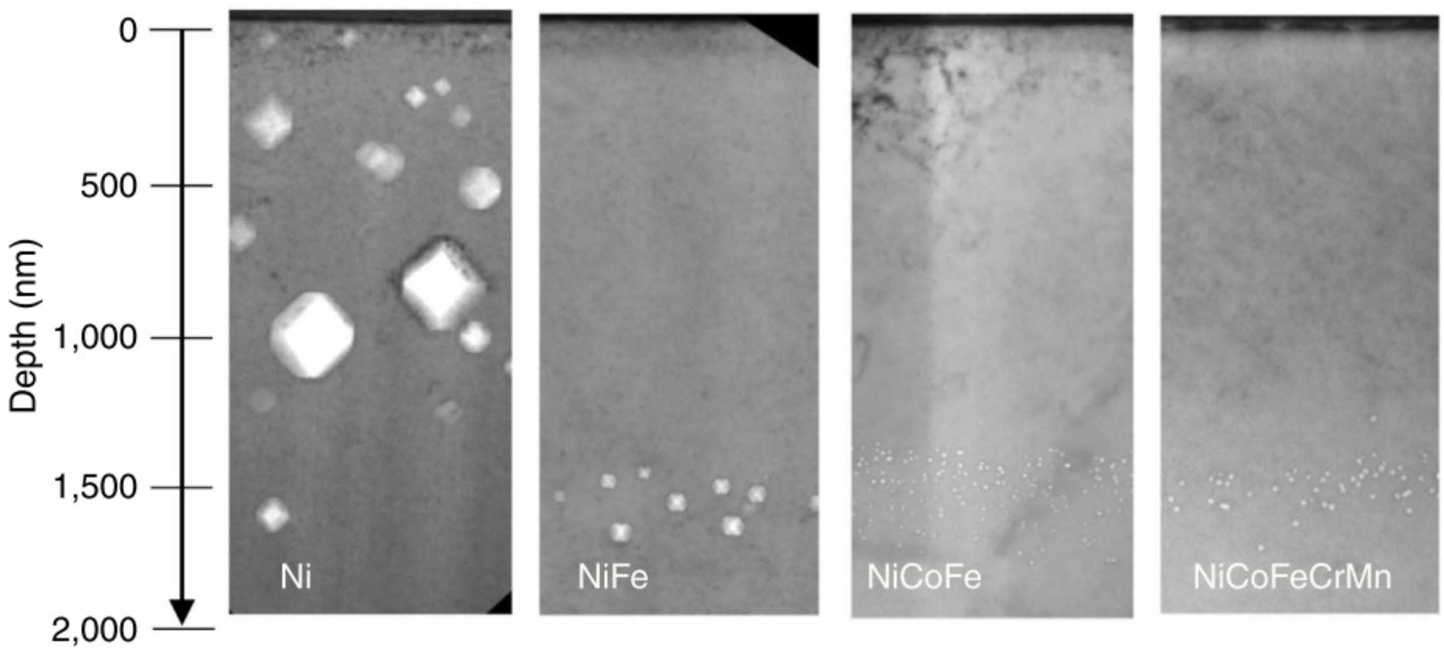


(a) Conventional alloy, racle, Senkov | 2017)

Swelling of increasingly complex alloys under ion irradiation (Jin | 2016)

Radiation Tolerance in HEAs

Both modeling and experimentation have shown HEAs can exhibit enhanced radiation tolerance <u>in the matrix</u>



3 MeV Ni^{+} ions
5×10^{16} cm^{-2}

T = 500 ° C

(Lu | 2016)

Design materials for extreme environments

ADDLab Alloy Design and Development

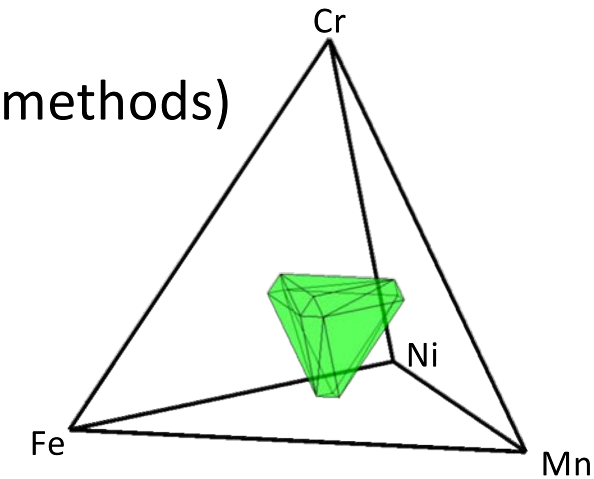
Processing Challenge: Develop materials with:

- Good chemical homogeneity
- Minimize processing defects
- Bulk samples (e.g., vs. thin film combinatorial methods)

High entropy alloys

Challenges:

- **Compositional space is large!**
- Thermodynamic calculations are exploring space with minimal data
- Microstructural control



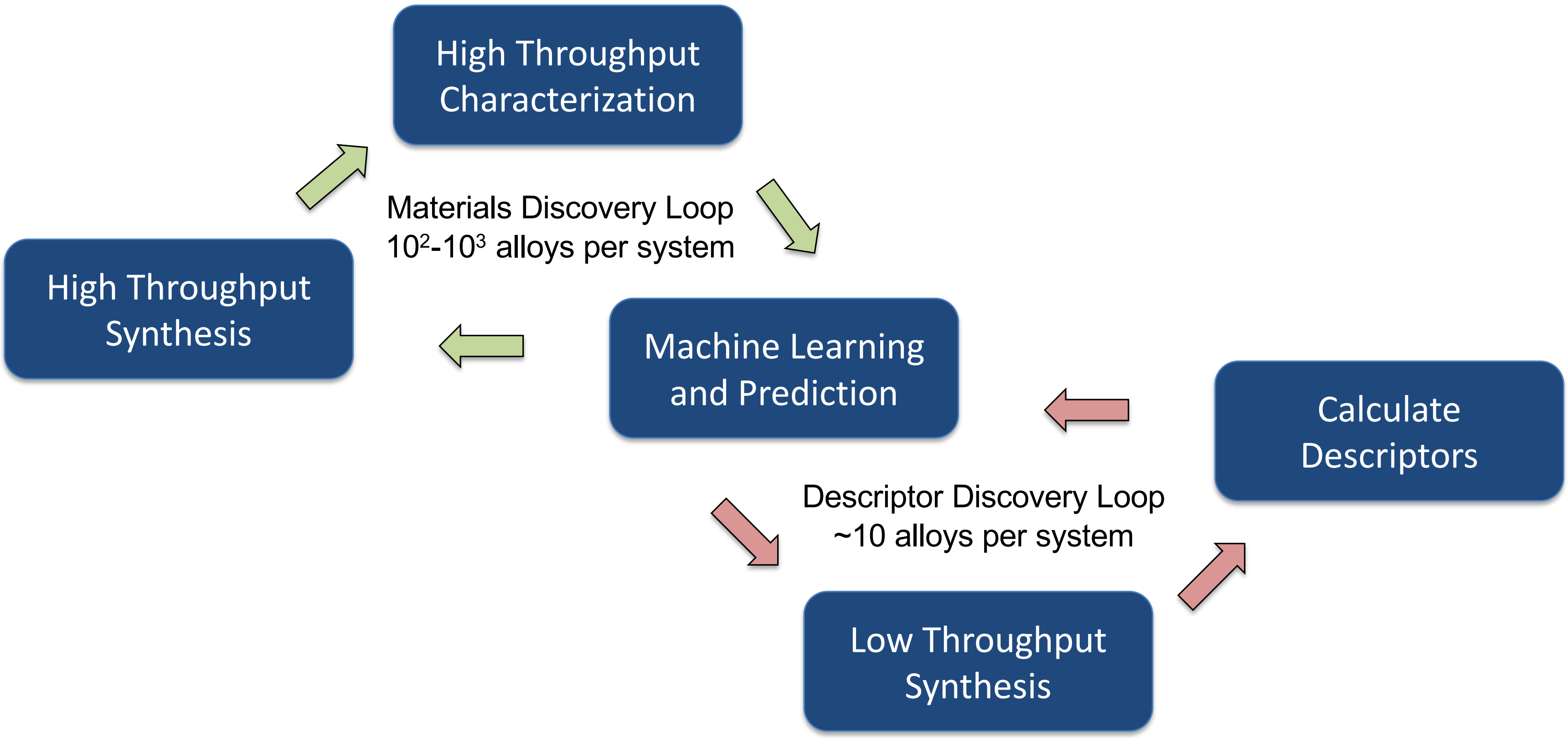
Each element varied between 5 - 85 at%

Need a high throughput experimental method!

Techniques for High-Throughput Discovey

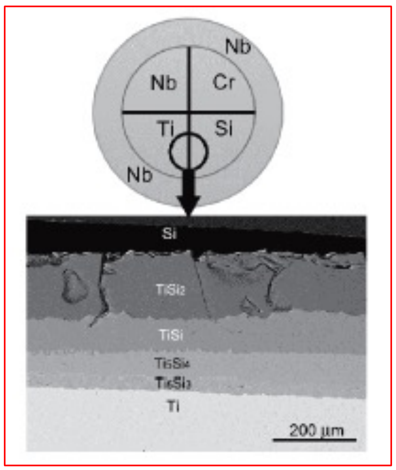
ADDLab
Alloy Design and Development

What are high-throughput experiments?



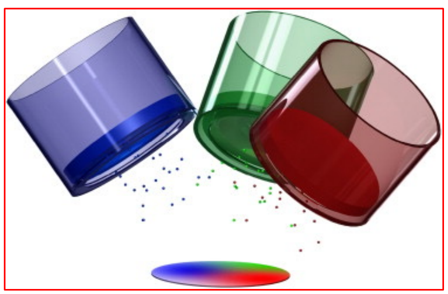
HTE Method	Advantage	Disadvantage	Image
Diffusion couples	Wide composition space	No bulk samples	(1)
Depositions	Great survey tool	Thin film, small composition regions	(2)
Functional grading with additive man.	Great survey tool	No bulk samples with same composition, powders	(3)
Arc-casting	Good bulk samples	Slower of the techniques, post-processing characterization	(4)

(1)



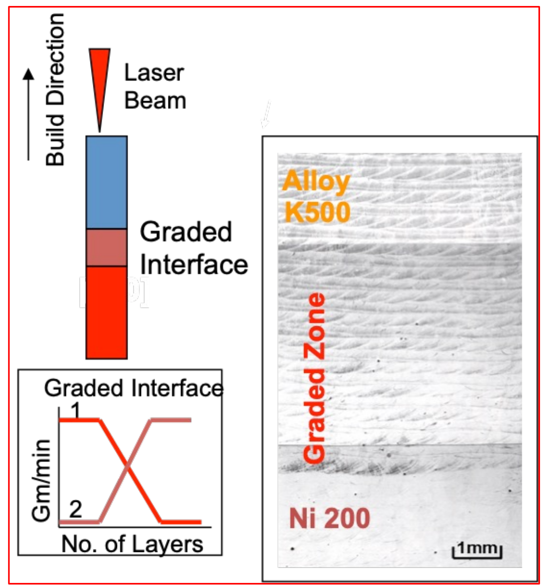
Zhao J C,, et al. MRS Bull, 27: 324–329 (2002).

(2)



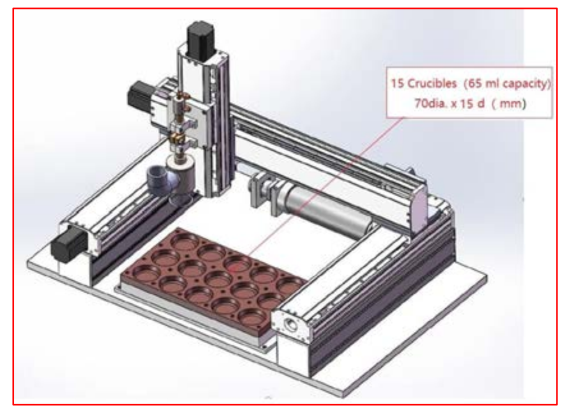
Gebhardt T. et al., Thin Solid Films, 520 5491 – 5499 (2012).

(3)



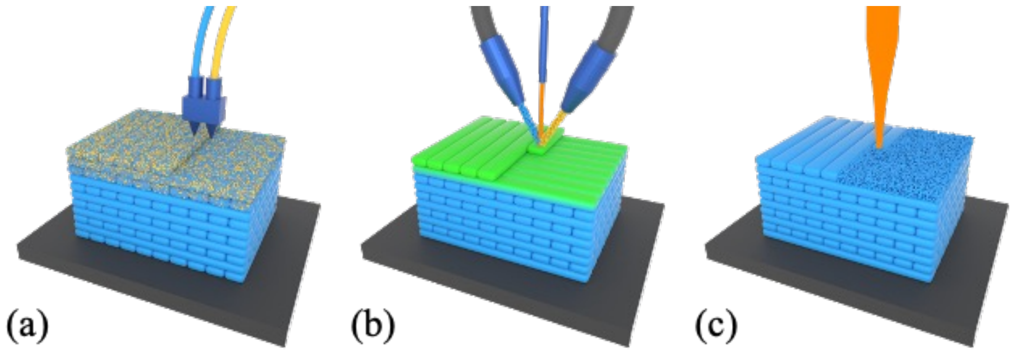
Hochanadel, P et al. Welding in the World 56 51-58 (2012).

(4)



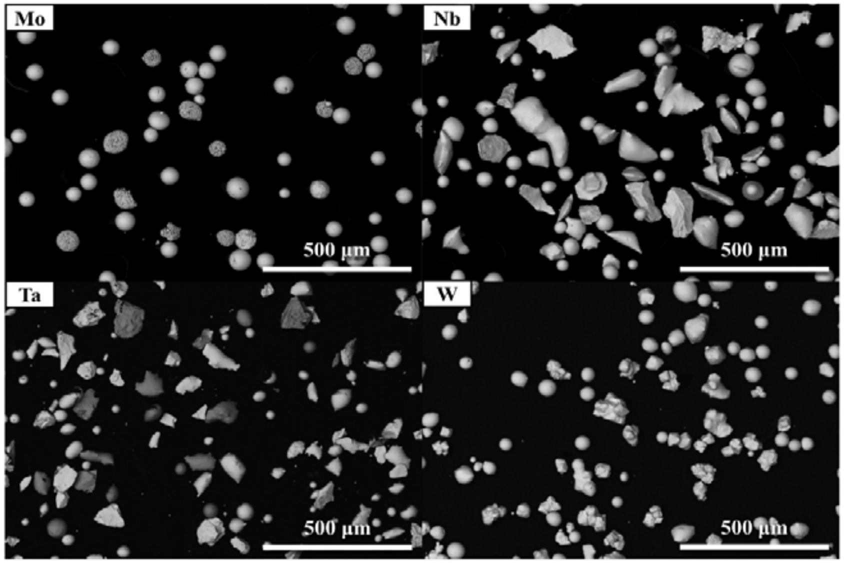
https://www.mtixtl.com/EQ-SP-MSM360.aspx

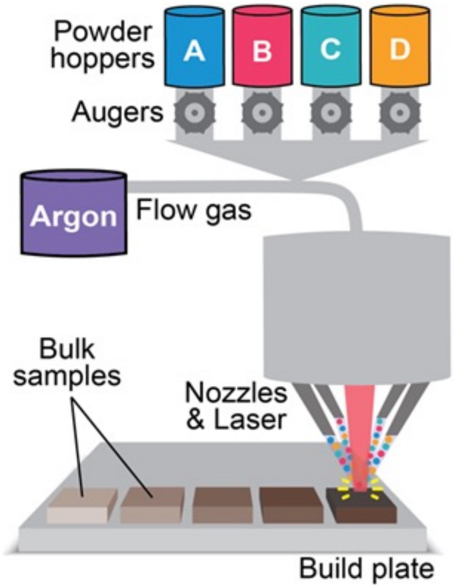
Viable additive manufacturing techniques for HT

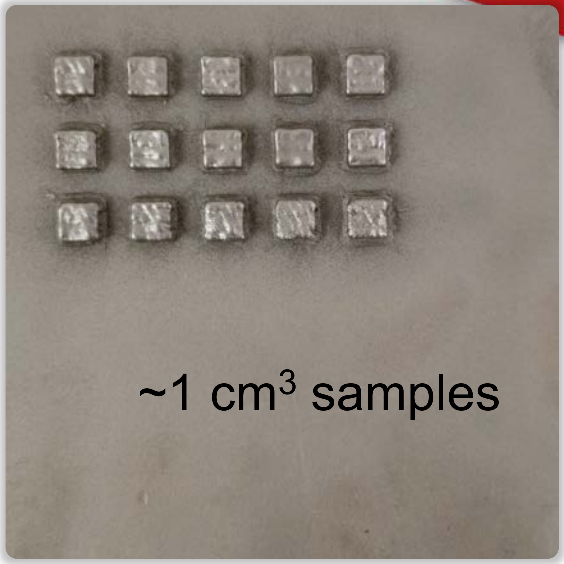


Technique	Pros	Cons
FDM (a)	• Flexible alloying • Fine dimensional features and surface finish • Functional grading is possible	• De-binding/sintering required • Distortion on sintering • Full density is difficult
DED (b)	• In situ alloying from most elements within 5 at.% • Densities >99% • Functional grading is possible	• 2.5 D vs 3D • Surface finish ~ 50 µm
LPBF (c)	• Industry preference for 3D • Densities >99% • Surface finish ~ 5 µm, Channels ~600 µm • Highest cooling rate (10^6 K/s), ~1 µm solidification segregation spacing	• Minimal material choices • No functional grading • Anisotropy of properties as a function of build direction

In situ alloying via directed energy deposition (DED)





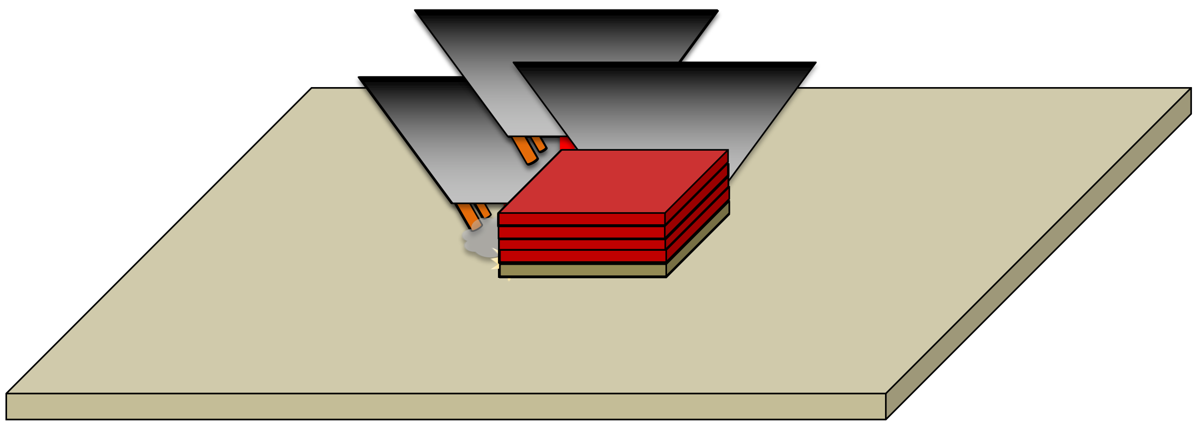


- DED can be used to rapidly fabricate bulk alloy samples via in situ alloying.
- Elemental powders are controlled independently.
- Powders are delivered to print head by argon flow gas.
- Laser down optic axis melts powders.
- 25-50 unique alloys can be synthesized in 4-5 hours (depends on required sample size).

Moorehead M, et al. Materials & Design. 2020;187:108358

Bulk HEA sample production via DED

- To produce printed stubs, a powder composition is selected and flown into the path of the laser, as it rasters across surface.
- Following material deposition, one or more remelting passes is performed to homogenize material.
- Process is repeated for five build layers to distance from build plate.



Challenges:

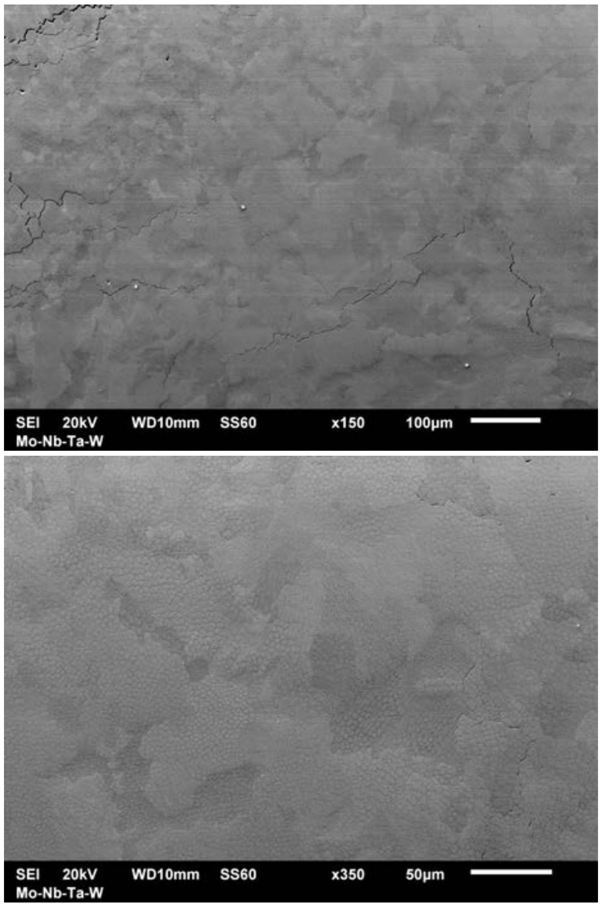
- Structural integrity of the sample stubs
- Unmelted powders
- Getting desired compositions in the 3D printed stubs

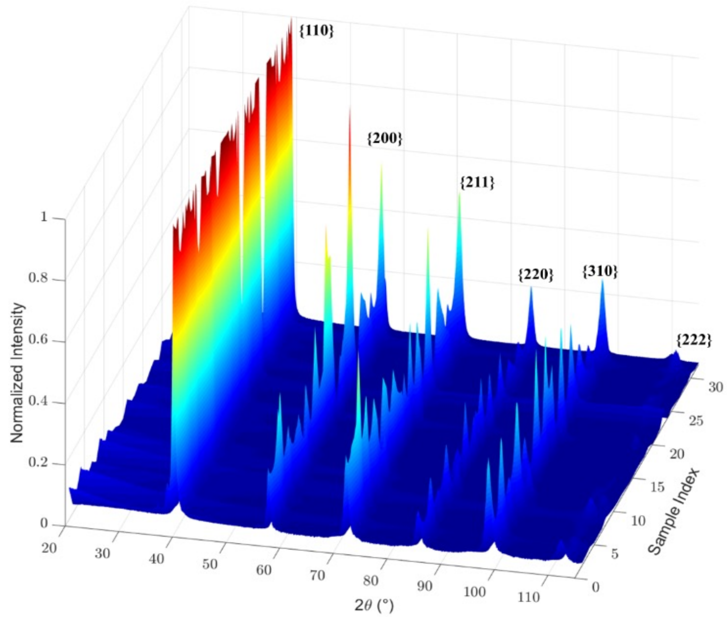
Matlab code developed by Michael Niezgoda controls:

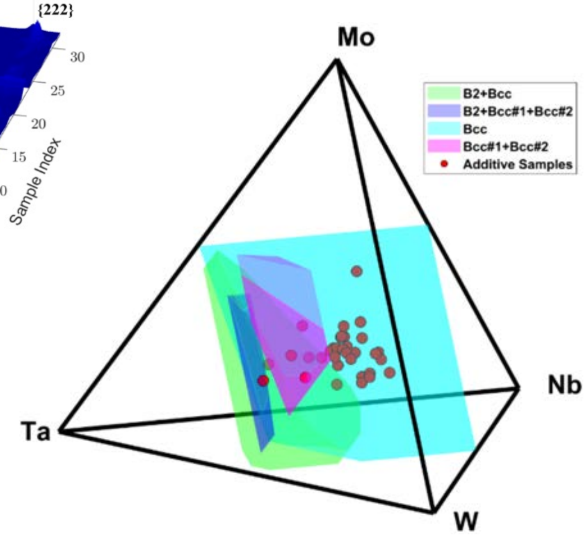
- Powder hopper RPM
- Laser power
- Laser head motion

Microstructural Characterization

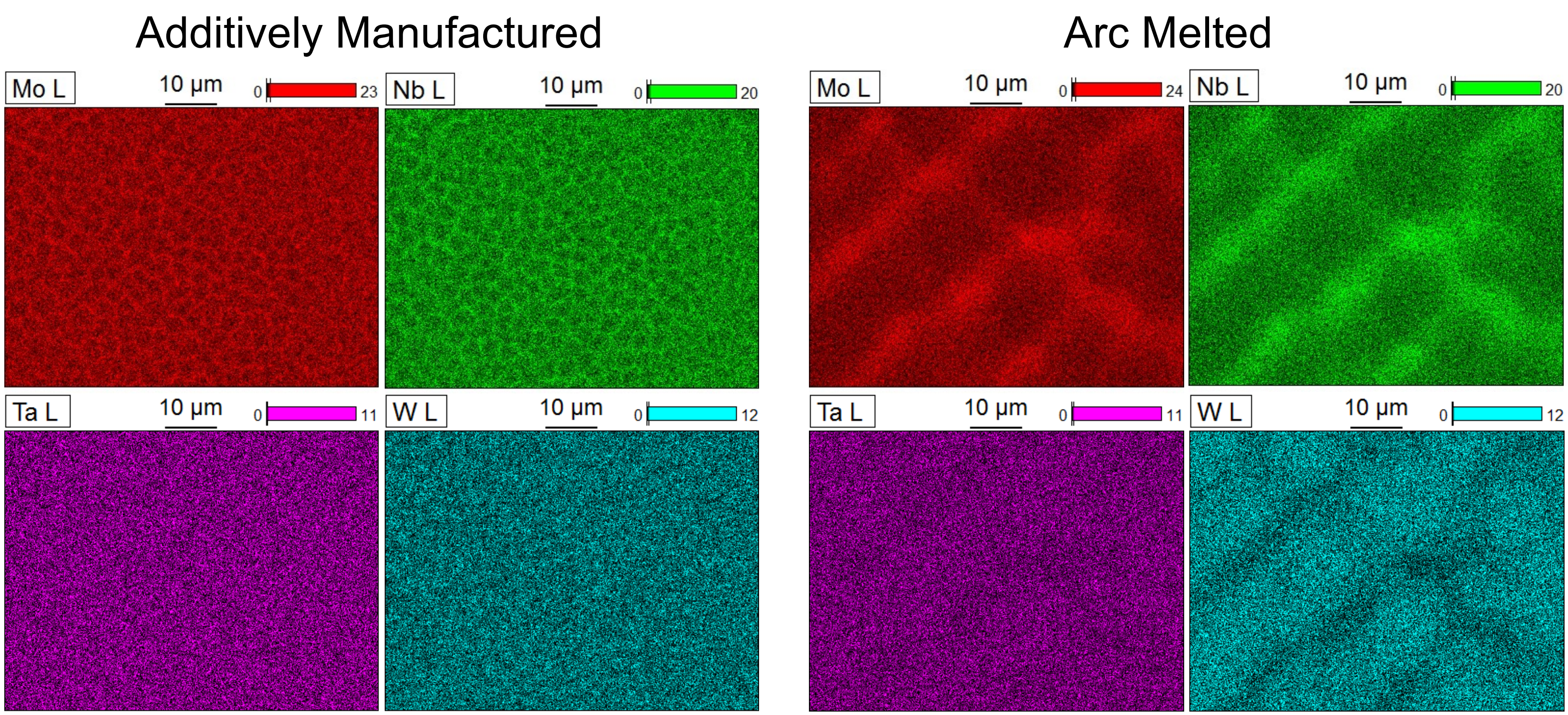
Additively Manufactured Equimolar MoNbTaW







Comparison with Arc Melting

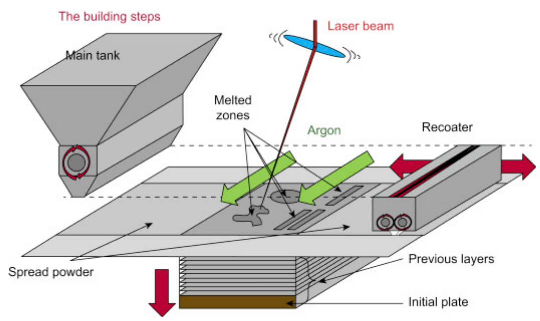


Scale-up for Commercial Product

ADDLab
Alloy Design and Development

Bringing HEAs from Research to Production

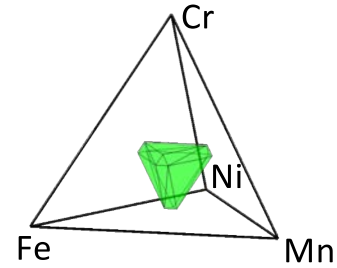
ADDLab
Alloy Design and Development

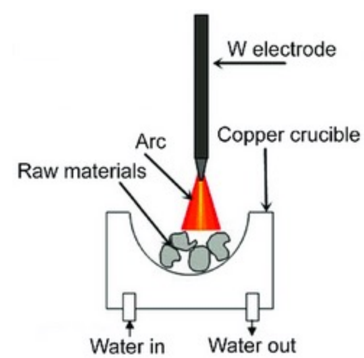


S. Sun, M. Brandt, M. Easton. "Powder bed fusion processes: An overview." https://www.sciencedirect.com/science/article/pii/B9780081004333000026

PBF Optimization and Production

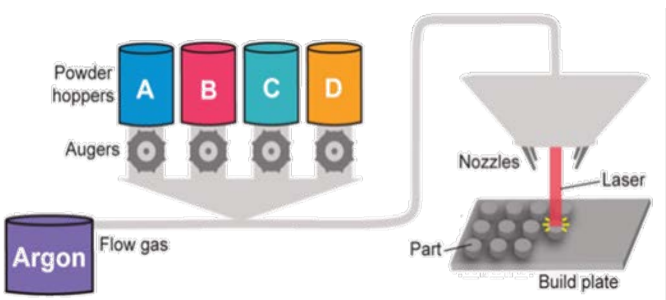
Modeling Predictions





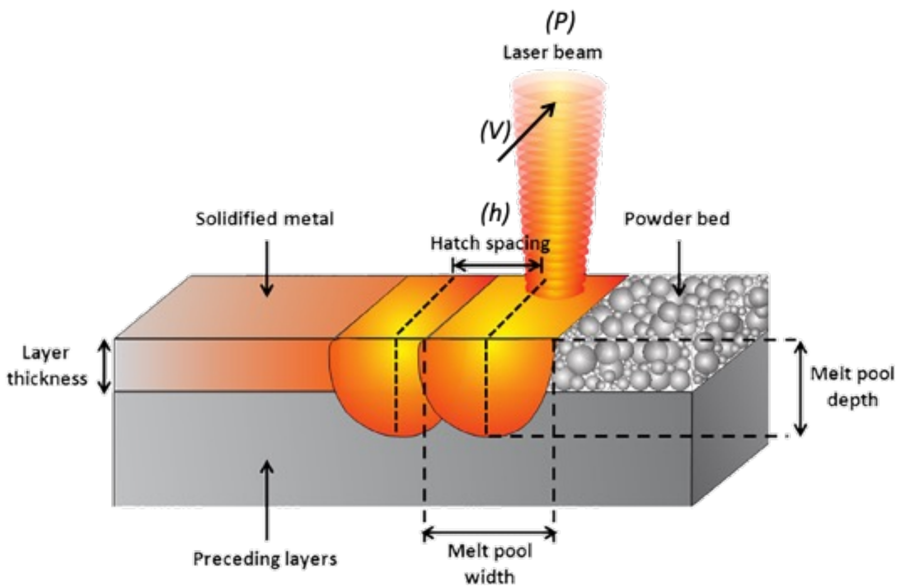
Verification with bulk Arc-Casting

High Throughput Testing with DED

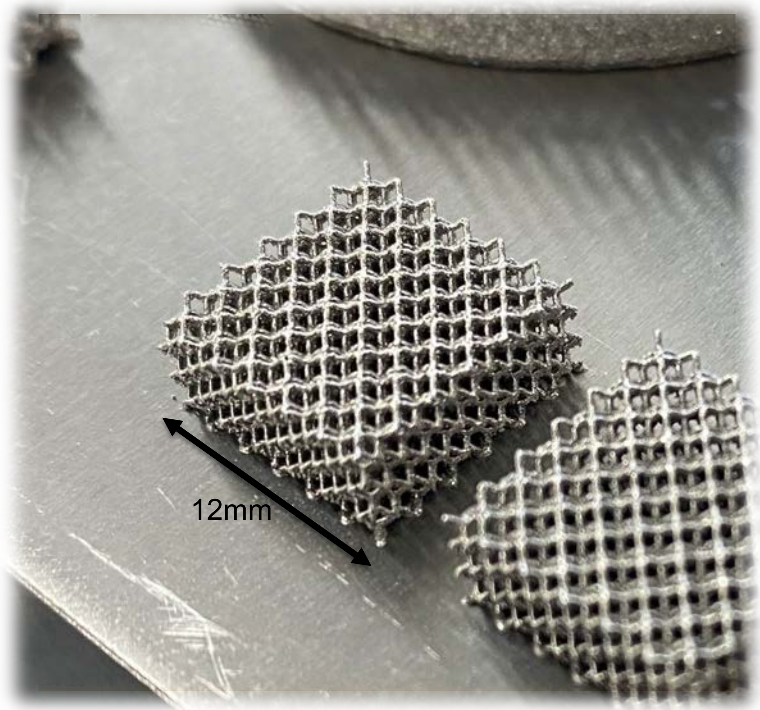


Simbarashe Fashu, Mykhaylo V Lototskyy, Moegamat Davids, Lydia Pickering. "A review on crucibles for induction melting of titanium alloys." https://www.researchgate.net/figure/Figure-S2-Schematic-diagram-of-the-vacuum-arc-melting-furnace-with-non-consumable_fig9_336984599

LPBF is a viable technology for scale-up of many advanced material

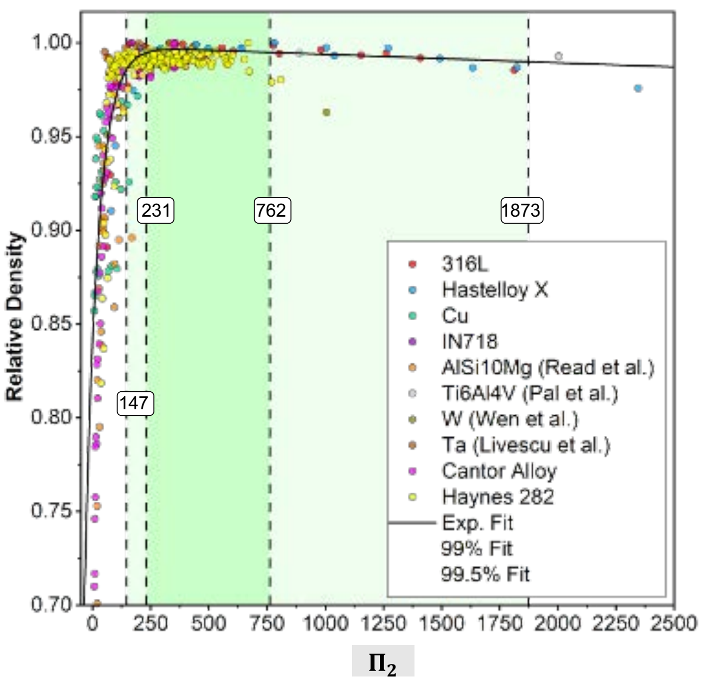


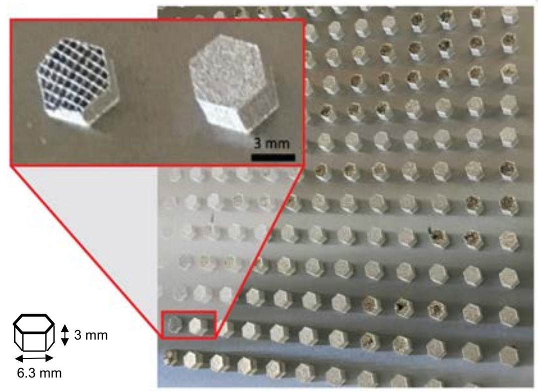
LPBF of W lattices



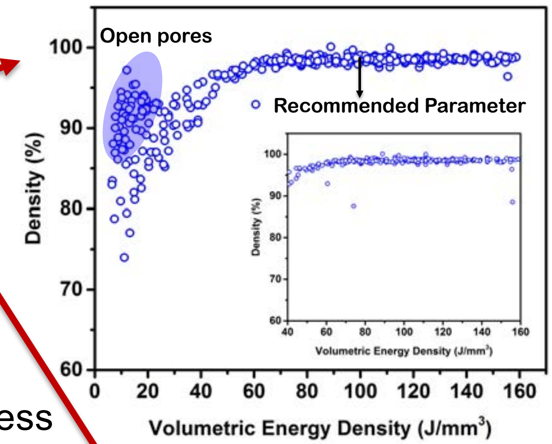
How do you design process parameters for AM, particularly for new materials? -----> LPBF

design matrix of process parameters



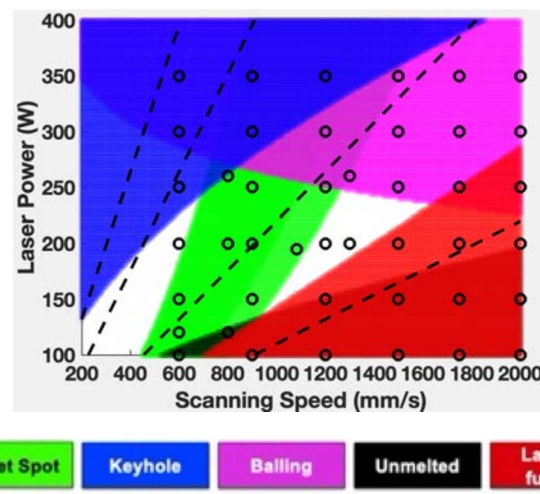


density

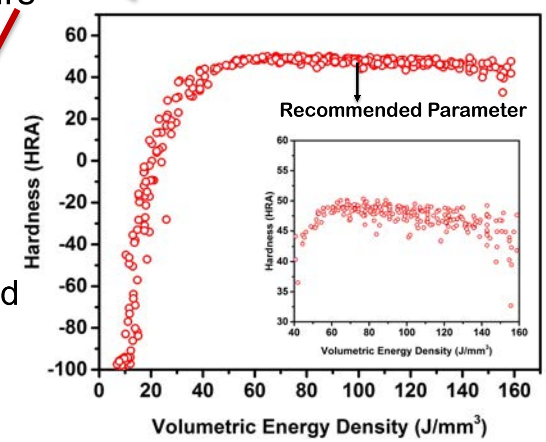


Hardness

16 hours

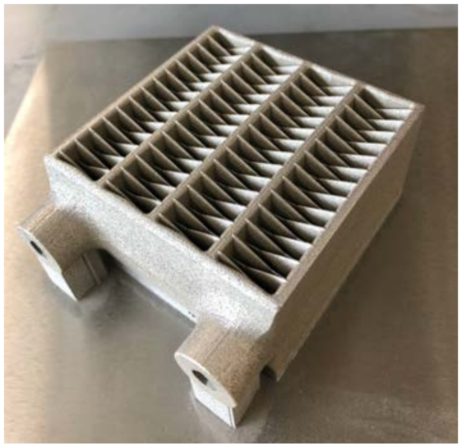


calculated process maps

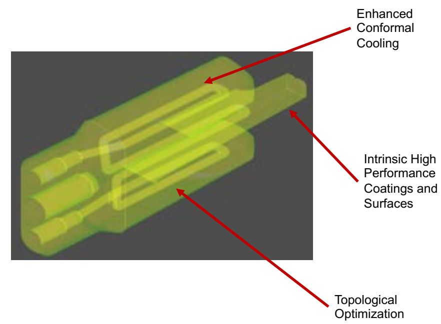


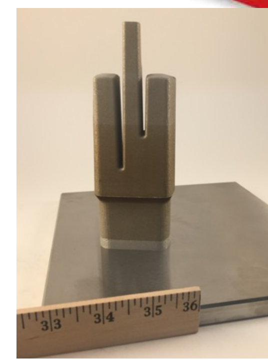
A.K. Agrawal et al. *MSEA* **A793** 139841 (2020).

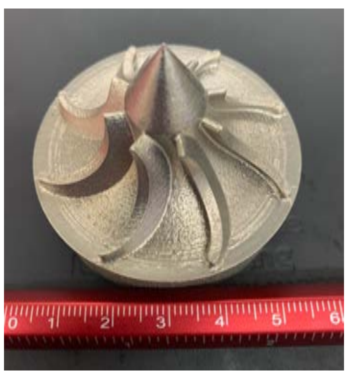
Examples of parts made with Industry



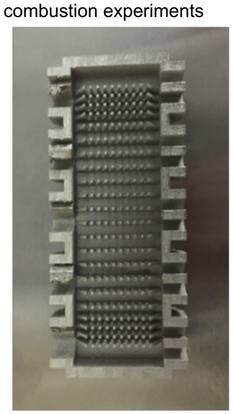
- Molding inserts
- Heat exchangers
- Turbines
- Hydrogen burners

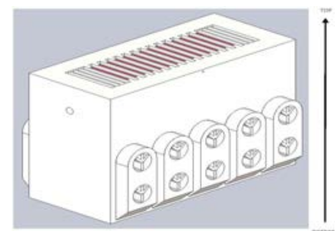


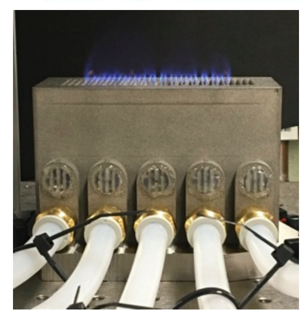




A burner for increased temperatures of hydrogen flames to perform combustion experiments



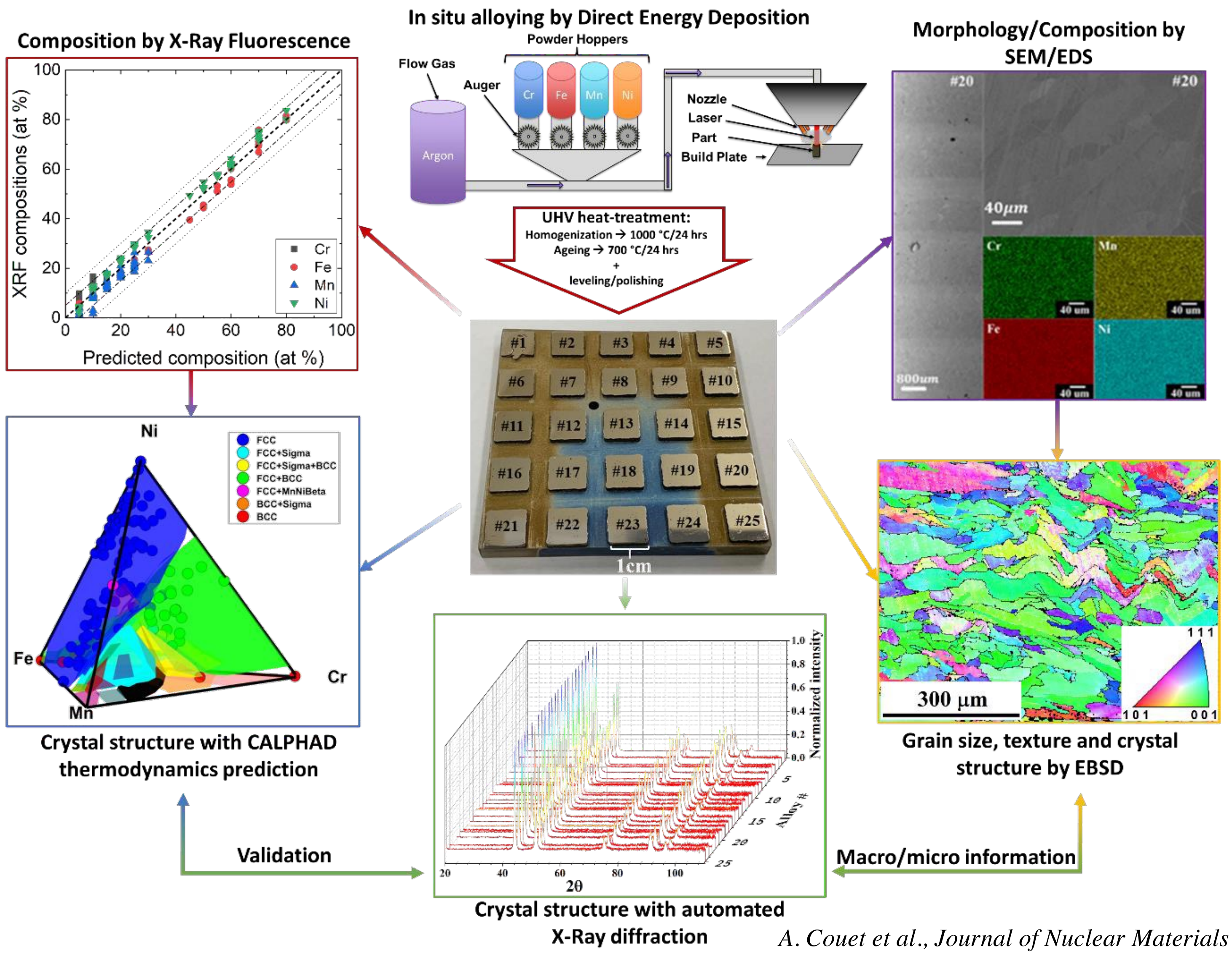




Challenges/Opportunities

Challenges/Opportunities

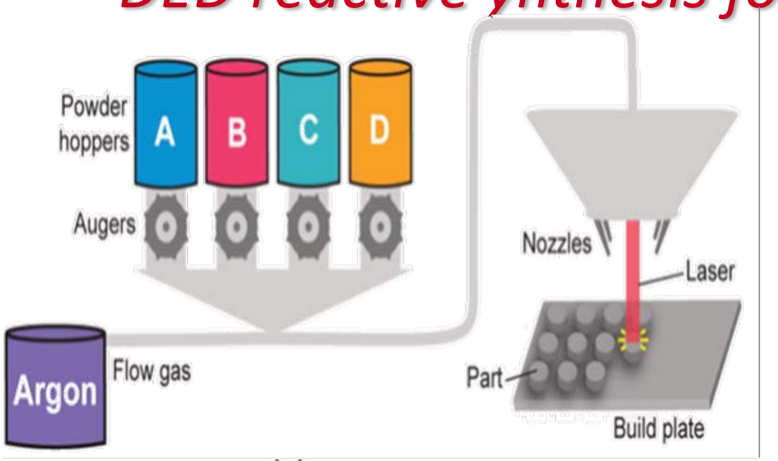
- Matching synthesis and characterization rates
- Feedstock availability and cost
- Process parameters/defects
  - Uncertainty quantification
  - Fatigue
- Scalability between AM techniques

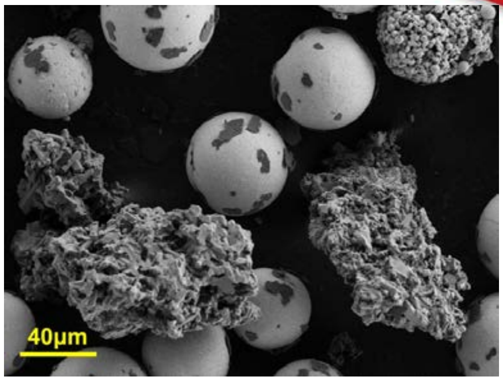


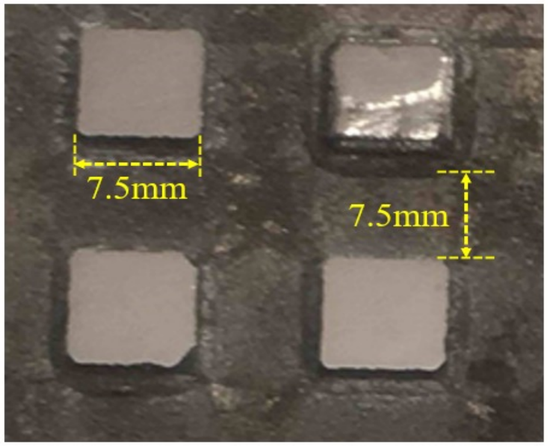
A. Couet et al., Journal of Nuclear Materials 559 (2022) 153425

Challenges and Opportunities: Feedstock

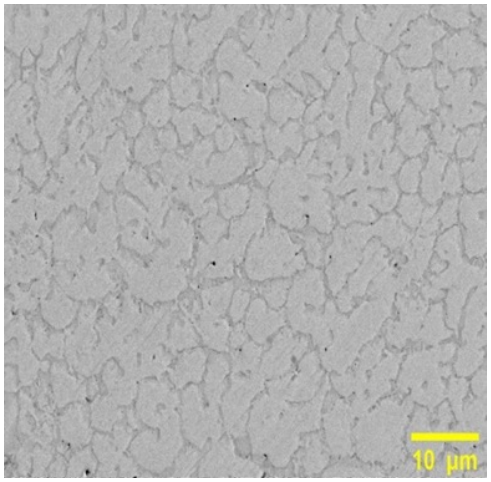
DED reactive ynthesis for reduced costs



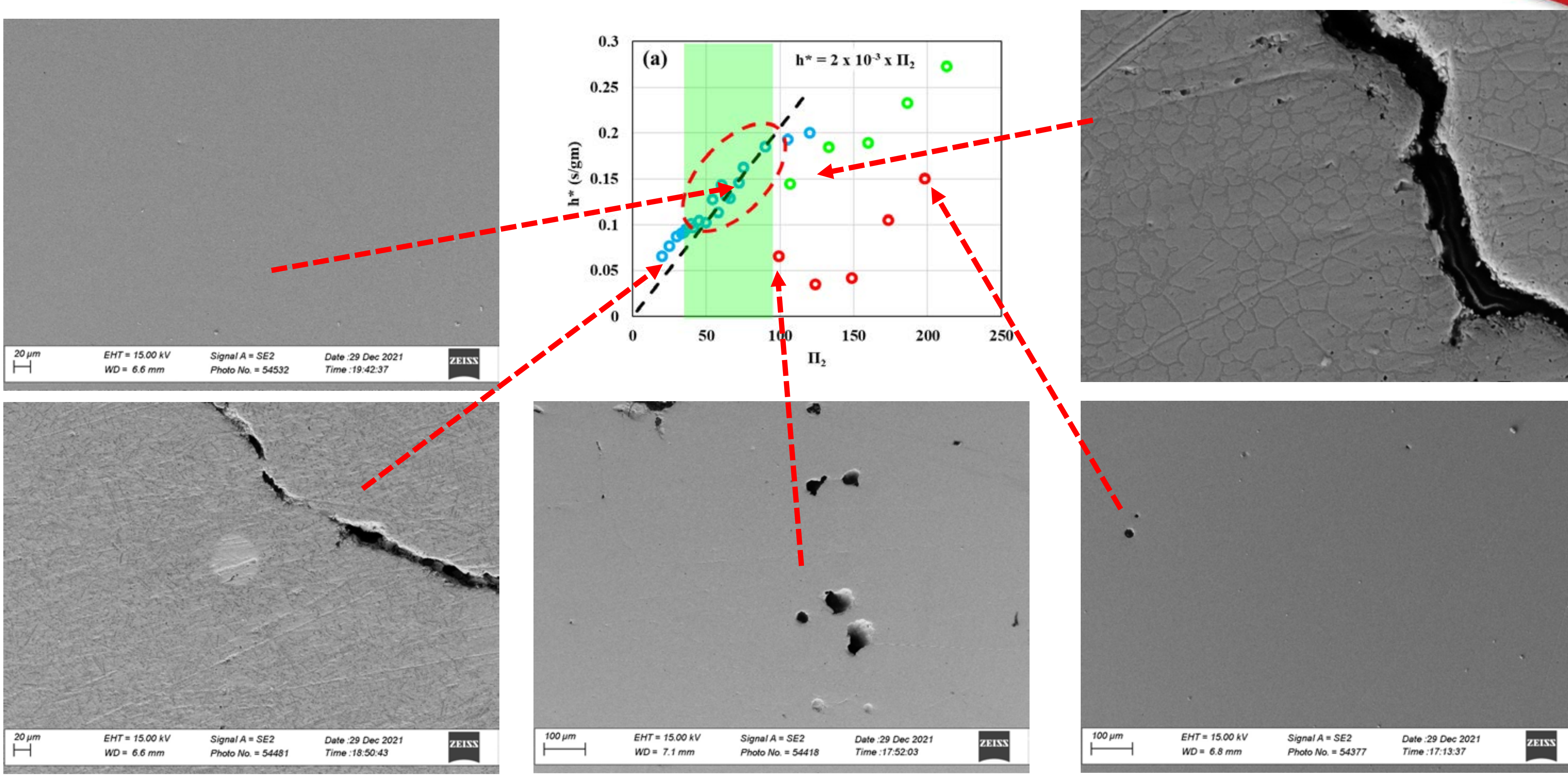




$$5Mo + 1/3Si_3N_4 + 2BN \rightarrow Mo_5SiB_2 + 5/3N_2$$

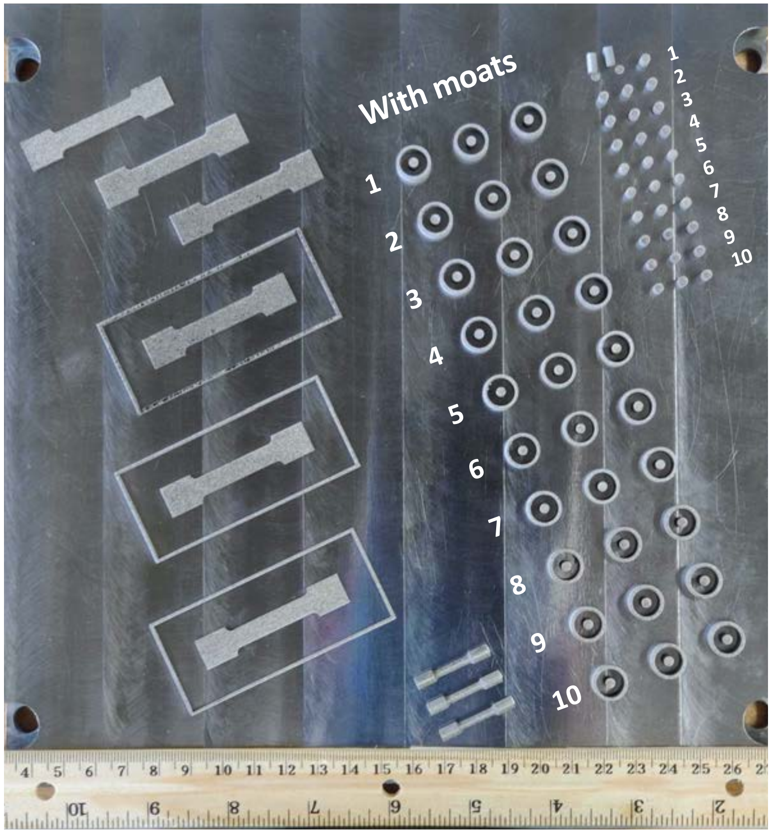


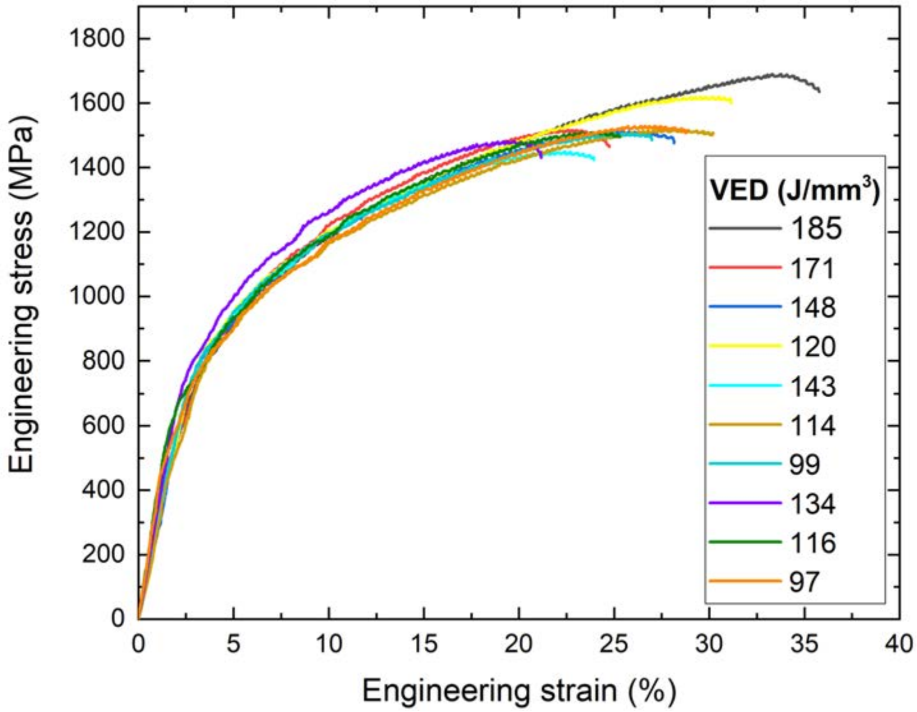
Application of dimensionless number to a Mo-alloy



Challenges and Opportunities: Feedstock

LPBF reactive synthesis for reduced costs





Discussion topics in summary

- Materials to consider
  - ODS, ceramics, W/W alloys, Carbon based composites, refractories, HEAs
  - Combinations of the above
- Advanced fabrication techniques for novel alloys
  - A few viable techniques, depending upon goals, but ultimately need bulk samples
- Scale-up potential
  - AM has currently demonstrated production capabilities
- Challenges/opportunities at production level for rapid design
  - Feedstock, process parameters, defects/cracking, scalability between AM techniques